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SYNTHESIS OF Y-Al GARNET

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An efficient new method of YAG synthesis is considered. The high efficiency of YAG production technology using a plasma torch for melt homogenization with subsequent crystallization and annealing is demonstrated.

Owing to expansion of the application of special synthetic minerals in electronics and other sectors of industry, the problem of their synthesis is quite important.

Application of yttrium-aluminum garnet (YAG) in quantum electronics is increasingly popular. YAG crystals, for example, are used as the elemental base for lasers. In recent years YAG has been successfully used in jewelry. Owing to its high transparency, dispersion, and bright luster, YAG is considered as an acceptable imitation diamond [1]. YAG has the structure of garnet and exhibits high hardness, chemical stability, and mechanical strength. YAG has no natural twin, therefore, the development of effective methods for its production is currently of great importance. A difficulty in YAG production is related to the high temperatures of synthesis (about 1970°C) [2]. Only non-traditional power sources, such as low-temperature plasma and laser beams make it possible to attain superhigh temperatures (2000°C and more) in a short time.

Our investigation included YAG synthesis (composition $3Y_2O_5 \cdot 5Al_2O_3$) in low-temperature plasma. YAG was synthesized in the argon plasma jet of a UPU-8M electric arc plasma gun. The initial materials were yttrium oxide ITO-2 (TU 48-4-191–72) and aluminum oxide (TU 48-5-58–73) whose granular composition ranged from 20 to 100 μm. The components in a stoichiometric ratio of 3 : 5 were mixed for 30 min in a ball mill with uralite balls in the ratio: material : water : balls = 1 : 1 : 5. The mixture obtained was dried, and the powder was moistened with water up to 5 wt.%.

The mixture was molded on a laboratory press into briquettes 15 mm in diameter and 20 mm high at a working pressure 30 MPa. The briquettes were placed in corundum crucibles of 0.1 and 0.5 liter capacity. The crucibles were coated with a mixture of milled chamotte and clay to protect them from the effect of abrupt temperature differences, molded into chamotte bricks, and fired in a muffle furnace at temperature of 950°C for 1.5 h. The GN-5p plasma burner of a UPU-8M plasma gun was used for melting the briquettes in the crucible. The plasma gun had the following parameters: working voltage of 30 – 32 V, current strength of 400 – 500 A. Argon was the plasma-forming gas, and its flow rate was 1.08 – 2.58 m^3/h at a pressure of 0.25 MPa. The water consumption for chilling was O.6 m^3/h.

In the first stage of YAG synthesis, the distance between the plasma burner and the briquette surface was 6 – 8 mm. Melting of briquettes proceeded for 10 – 15 min, and a melt was formed in the crucible. At the same time, the melt was subjected to intense homogenization by a plasma-forming gas current at a pressure of 0.24 – 026 MPa and gas flow rate of 2.28 – 2.58 m^3/h. The stage of homogenization of the emerging melt by the plasma-forming gas current is the crucial one, since it provides for a crystalline melt that is homogeneous over its entire volume. If the homogenization stage is not used in synthesis of YAG, the crystallization products in addition to YAG will contain $2Y_2O_3 \cdot Al_2O_3$ and $Y_2O_3 \cdot Al_2O_3$ anisotropic phases in the amount of 9 – 11% [3].

The method developed by us implies that a melt of high-melting oxides is homogenized with a heat-carrying gas whose jet dynamics are determined by the operating parameters of the plasma gun (pressure and gas flow rate). In this case, melting with constant stirring makes it possible to obtain a homogeneous melt and, later on, a homogeneous product, according to the prescribed stoichiometry.

In the second stage of YAG synthesis, the distance from the plasma burner exit section to the melt surface was 10 – 12 mm, with plasma-forming gas pressure of 0.18 – 0.21 MPa and flow rate of 1.08 – 1.50 m^3/h. It ought to be noted that homogenization facilitates the stoichiometric distribution of the mixture components in the melt. This develops the conditions for crystallization of the preassigned crystalline phase. Homogenization provides for fast dissolving of the initial components in the melt, because the dissolution process is shifted from the diffusion area to the kinetic area. In subsequent crystallization, the stresses in the crystallized product decrease significantly, owing to the existence

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of only one preassigned phase, and the yield of the end product increases. The conglomerate synthesized was annealed in a muffle furnace at a temperature of 1050°C for 1.5 – 2 h. Annealing contributed to partial removal of internal stresses in YAG.

In the course of YAG synthesis, it was necessary to determine the parameters of the plasma-arc torch of the UPU-8M unit. The average mass temperature of the plasma torch at the nozzle exit section with current strength of 400 A and argon flow rate of 2.28 m^3/h was equal to 8644 K, and the ionization degree of the plasma-forming gas calculated by the Sag equation was 29% [3]. The density of the YAG was determined by the standard pycnometric method, and the microhardness was measured with a PMT-3 microhardness meter. The measurements were performed on polished sections. The microhardness was calculated from the expression in [4]

$$H = 18{,}188\frac{P}{d^2},$$

where H is the hardness number, MPa; P is the load at the diamond pyramid apex, equal to 150 g; d is the length of the diamond pyramid imprint diagonal, μm.

The calculated value of the YAG microhardness was 17037 ± 150 MPa, which corresponds to 8.5 on the Mohs scale. The optical characteristics were determined with a MIN-8 polarization microscope according to GOST 26822–86 [4].

Immersion samples were prepared in the following way. YAG crystals were crushed and milled in an alundum mortar with an alundum pestle (hardness on Mohs scale of 9, H = 20,209 MPa) to grains 30 – 60 μm in size. A batch of 30 – 40 mg was placed in the center of a clean laboratory table and covered with a cover glass (area up to 1.5 cm^2) under which a drop of immersion liquid was immersed.

In this way samples were prepared with various immersion liquids having different refractive indexes. The essence of the method is observation of the Becke line emerging at high magnification in the form of a light stripe at the boundary of two transparent media. The index of refraction was determined using immersion liquids with refractive indexes of 1.74 – 2.06. The internal stresses in the YAG crystals were measured on a PSK-250 polariscope/polarimeter [5]. The results of the investigation are given below:

Properties and Parameters of YAG Synthesis

Granular composition of initial components, μm	20 – 100
Time, h	
of synthesis	0.5 – 0.6
of annealing	1.5 – 2.0
YAG density, g/cm^3	4.15
Refractive index	1.87 – 1.88
Hardness on Mohs scale	8.5
Stresses in end product, MPa	2 – 3
Waste in cutting the conglomerate, %	30 – 40

The method of YAG synthesis proposed here is more efficient than the classical Verneuil method involving a similar technical solution [1].

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